

Portland Harbor Sample Receipt, Analysis, and Results Report

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Introduction

This report is part of a baseline fish tissue study at the Portland Harbor Superfund Site (Portland Harbor Site). The U.S. Environmental Protection Agency (EPA) conducted this study as part of the Portland Harbor Remedial Investigation and Feasibility Study (RI/FS) and in conformance with the National Contingency Plan. The City of Portland provided assistance at EPA's request.

This study adds to the body of information that will be used to establish baseline concentrations of polychlorinated biphenyls (PCB) in smallmouth bass tissue. EPA will use the updated baseline data as a point of comparison to future contaminant concentrations measured in smallmouth bass during and following remedy implementation. The area being investigated is referred to as the Portland Harbor Study Area (study area).

This report summarizes sample receipt, analysis, and results of fish tissue samples collected during the baseline fish tissue study. The samples were collected in accordance with a sampling and analysis plan (SAP) prepared for the EPA by GSI Water Solutions, Inc. (GSI) (GSI, 2011), and as documented in the Field Sampling Report (GSI, 2012).

The report includes three main sections. The first section discusses the receipt of samples by EPA, subsequent sample processing, and sample shipment (to and from the analytical laboratories). The second section discusses the analysis of samples at each laboratory and provides a summary of data validation results. The final section discusses the sample results, including a discussion of data reduction steps used to calculate summed totals to be used in the RI/FS and risk assessments (RA). Tables of results and the data validation report are attached to this report.

This report was prepared by Tetra Tech under EPA contract number EP-W-07-078. Tetra Tech was responsible for subcontracting laboratory analysis to Pace Analytical Laboratories (Pace), preparing the data for database entry, and preparing this report.

Sample Delivery, Receipt, Processing, and Analysis

As described in the Field Sampling Report, smallmouth bass were collected at 68 "Target" sampling locations and 14 "Lifecycle" sampling locations. The Target samples were generally within the 225- to 335-millimeter (approximately 9- to 14-inch) size range specified in the SAP while the Lifecycle samples ranged in size from 197 to 455-millimeters. The Target samples were collected by GSI and shipped to KAP Technologies Inc. (KAP), laboratory in Woodlands, Texas, on September 19 and October 11, 2011. The Lifecycle samples were shipped to the U.S. Army Corps of Engineers (USACE) Engineer Research and Development Center in Vicksburg, Mississippi, on October 11, 2011. Table 1 identifies the samples as either Target or Lifecycle and indicates their respective shipment batch. Figure 1 shows the locations of the samples collected. More detailed location maps are included in the Field Sampling Report prepared by GSI.

The initial shipment of Target samples, 50 in all, was incorrectly processed by KAP. The samples should have been homogenized as whole body samples and divided into two separate aliquots. One aliquot was intended for analysis of semivolatile organic compounds (SVOC), polycyclic aromatic hydrocarbons (PAH), and organochlorine pesticides by KAP, and the other aliquot was to be shipped to Pace in Minneapolis, Minnesota, for analysis of PCB congeners and lipid content.

Instead of following contract specifications, KAP prepared skinless fillets and discarded the skin and carcass portions. Upon discovery of this mishap, EPA determined that the best course of action was to reframe from analyzing the skinless fillet samples and that the 18 unprocessed Target samples held at KAP and the 14 Lifecycle samples held at the USACE Vicksburg facility should be shipped to EPA's Manchester Laboratory (MEL) in Port Orchard, Washington. These 32 samples then were divided into fillet (skin on) and carcass portions, weighed, homogenized, and shipped to Pace for analysis of PCB congeners and lipid content. Fillet removal was conducted in accordance with the procedure described in Appendix C of the *Portland Harbor RI/FS Round 3B Field Sampling Plan for Fish and Invertebrate Tissue and Collocated Surface Sediment* (Integral, 2007). The skin and belly flap were included in the fillet sample. Because of budget restrictions, modifications to the processing scheme, and termination of the KAP contract, EPA determined to forego the analysis of SVOC, PAH, and pesticide. Table 1 summarizes the samples collected, and identifies which samples were not analyzed as a result of the processing error. The chains of custody and sample processing forms are included in Appendices B and C to this report.

Pace analyzed each sample for the following analyte groups:

- PCB following SOW SOM01.2 and EPA Method 1668C
- Lipids following SOW SOM01.2 and EPA Method 1668C

Following analysis and data validation, the remaining sample material was returned from KAP and Pace to MEL for long-term archival.

Data Validation

EPA Region 10 quality assurance (QA) staff validated the data following *Guidance on Environmental Data Verification and Validation* (EPA, 2002), *EPA Contract Laboratory Program (CLP) National Functional Guidelines for Organic Data Review* (EPA, 2008), and *EPA Region 10 Standard Operating Procedure (SOP) for the Validation of Method 1668 Toxic, Dioxin-like PCB Data* (EPA, 1995). A Stage 4 data validation was conducted on all analytical data (EPA, 2012).

Table 2 summarizes the results of the data validation (see tables attached to this report). The data validation report is included in Appendix B to this report.

EPA made few data qualifications during the data validation process. Qualifications were made for method and proof blank contamination, chromatographic interferences, and chromatographic peak saturation.

Data validation included an assessment of blank samples, including four rinsate blank samples collected during sample homogenization. Several analytes were qualified as estimated or non-detect based on trace laboratory blank contamination. The rinsate blank samples were analyzed for PCB congeners, and the results were compared with associated samples. Although a few congeners were detected at trace levels, EPA did not qualify any samples based on rinsate blank contamination because no detected sample results were detected at concentrations within 5 times higher than the value in the associated rinsate blank (EPA, 2012).

Sample Results

In addition to the raw results, the SAP (GSI, 2011) specifies two sets of rules for summing data and retaining or modifying qualifiers and reducing the data to a single value per sample and summation group. The two sets of rules include one for use in the RI and another for use in the RA and background data sets, in accordance with the Portland Harbor RI/FS guidelines (Kennedy/Jenks, 2004).

Summation rules for the RI data set are as follows:

- For samples with at least one detected result for the summed analytes included in the total:
 - Detected concentrations are included in the calculated total.
 - Non-detected concentrations are not included in the calculated total (i.e., treated as zero).
- For samples with no detected results:
 - The highest detection limit is used for the summation. The calculated total result is indicated with a “U” to indicate it was not detected.

Summation rules for the RA and background data sets are as follows:

- For samples with at least one detected result for the summed analytes included in the total:
 - All detected concentrations are included in the calculated total.
 - All non-detected results for analytes, if they were detected at least once in the RA data set within the study area for a given medium (in this case, fish tissue), are included in the calculated total at one-half the detection limit.
 - All non-detected results for analytes, if they were not detected in any sample within the RA data set within the study area for a given medium, are not included in the calculated total (i.e., treated as zero).
- For samples with no detected results, where some of the summation analytes are determined to be present within the study area:
 - The highest detection limit for analytes present within the study area is used for the summation. The calculated total result is indicated with a “U” to indicate it was not detected.

Data qualifiers were carried through the summation procedure. If all the analytes were not detected, a “U” qualifier is applied to the summed data to indicate that all results were reported as not detected. All calculated totals are flagged with a “T” qualifier to indicate that they are mathematically derived values.

After the summations were complete, an overall concentration was calculated for each fish sample. These concentrations are reported as “whole body (calculated)” in the tissue field. The whole body concentrations were calculated only for the summation analytes (and not including the homolog summations), and were calculated using the following equation:

$$C_{wb} = \frac{(C_f \cdot M_f) + (C_{bwof} \cdot M_{bwof})}{(M_{fillet} + M_{bwof})}$$

where:

C_{wb} = the calculated concentration of the whole body fish sample

C_f = the measured concentration of the fillet fish sample

C_{bwof} = the measured concentration of the body without fillet fish sample (i.e., carcass)

M_f = the mass of the fillet fish sample

M_{bwof} = the mass of the body without fillet fish sample

Table 3 lists the summation group results to be calculated for each sample. Table 4 summarizes the laboratory data, including the summed totals as defined in Table 3 and the summation rules listed above.

References

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EPA. 2012. Memorandum. Subject: Data Validation Report for the full list of 209 Polychlorinated Biphenyl Congener (PCB Congeners) Analyses of Small-Mouthed Bass Tissue Samples Collected for the Portland Harbor RI/FS BACE Project Numbers: 10180829, 100180826, 101867, 101870. From: Ginna Grepo-Grove, R10 QA Manager, Office of Environmental Assessment, USEPA. To: Chip Humphrey and Matthew Lambert. June 13, 2012.

Figures

Tables

Appendix A

Chain of Custody Forms

Appendix B

Data Validation Report